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Multiscale dimensional tolerance specifications established on shrinkage assessment in ceramic micro injection molding

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Abstract

Taking advantage of a micro powder injection molding production of a critical functional component in a miniaturized mechanism, a method for the formulation of specification intervals was developed, based on the evaluation of the shrinkage.
The synthesis allows to allocate tolerance intervals according to a desired risk of rejecting parts in future evaluations. Such specifications are formulated as a function of the shrinkage, optimizing its impact on different features of size independently of their shapes or dimensions.
The method is of general validity for any molding process where the material undergoes a change in dimensions due to a phase transformation.

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Keywords: shrinkage; shrinkage uncertainty; tolerance interval formulation.

1. Introduction

The specifications of manufactured parts are essential requirements for defining their quality in accordance with the production. In addition, quality assurance has undoubtedly need of an adequate assessment of the measurement uncertainty and the establishment of traceability. They are both properties of the measurements that, not only depend on the techniques adopted for their assessment, but also on the measurement instrument.

Mutual dependences on uncertainty and traceability of production and measurement process variabilities have been deeply investigated in the past (e.g. [1]-[3]). The pragmatic *golden rule* of metrology recommends an uncertainty lower than 10 % of the tolerances under verification [1]. A number of errors and different influence factors, in fact, may affect different measurement instruments in a typical uncertainty evaluation. Beyond the *golden rule*, a measurement process uncertainty can be identified combined to the one of the production process and dealt with statistical techniques, e.g. process capability indices [2]. Hence, if the measurand is defined unambiguously according to the functional requirements, metrology becomes a powerful tool for gaining information and, consequently, generating knowledge for "making decisions" [3].

Nowadays, general indications to deal with the conformance verification can be found in official documents [4]-[5]. However, no official documents are available for addressing the inverse problem of the formulation of a specification zone. Nonetheless, several specific works exist in literature for allocating tolerances, even though predominantly for assemblies. They include different methods, from traditional ones (scaling, minimum-cost function, Lagrange multipliers, etc. [6]-[9]) to more recent ones (cost/risk estimation, fuzzy logic, specifications based on Monte Carlo method [10]-[13]).

The inverse problem of the formulation of specification intervals is subject of this paper. It proposes a method that is relevant and valid when it is possible to measure a shrinkage. Hence, the method is considered particularly suitable for microinjection molding technologies where, as a consequence of a phase transformation (melt to solid polymer), the dimensions of the master geometry undergo a change in the dimensions of the replicated substrate.

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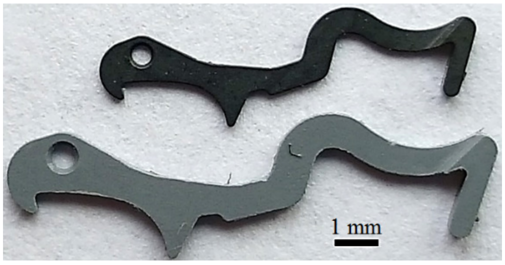

Fig. 1. Examples of final product (top) and intermediate component (bottom).

The method is presented and validated on components produced by micro-powder injection molding (µPIM). Such production has notoriously huge shrinkage with respect to the master geometry.

Furthermore, defects on edges are very common on the final parts. As a consequence, there are some limitations related to the quality control [14], so that such components are especially suitable for introducing the method.

In other injection molding technologies other factors may influence the achievement of the desired dimensions, e.g. a non-complete replication. Nonetheless, they cause a similar shortage in the achieved dimensions, which can be quantified with the same method for the formulation of tolerance intervals, comparing the production to the related master geometry.

Past works already investigated the µPIM dimensions replication [15], [16]-[17], the achievable surface topography [15], [18]-[20] and the influence of molding parameters on dimensional accuracy [15], [21]. Conversely, in the current study, taking advantage of a specific study case, the proposed method addresses the formulation of specification intervals, as a function of the shrinkage, considering the following aspects: 1) Impact of the shrinkage on the dimensions. 2) Conformance verification of the specifications stated by the manufacturer. Nevertheless, the purpose is to introduce the method. Hence, a complete investigation on product conformance with specifications at the micro scale can be found elsewhere [22].

2. Parts manufactured by ceramic injection molding

Micro-powder injection molding is considered an interesting manufacturing process for complex micro parts or micro structured parts. In fact, i) miniaturized manufacture at a relatively low cost, ii) chances to have mass production and, finally, iii) assembly steps integrated into the process (co-injection and co-sintering) turn µPIM into a particularly attractive technology [14]-[15], [23].

In depth, intermediate parts (commonly called green parts) were obtained by ceramic injection molding (CIM). The process was performed by an Arburg Allrounder 270 S 250-60, with a diameter of the reciprocating screw of 15 mm, a diameter of the nozzle of 2 mm and a maximum clamping force of 250 kN.

The ceramic feedstock used for CIM was Catamold® TZP-F 315 produced by BASF SE, i.e., a compound of zirconium dioxide (ZrO_2), stabilized by diyttrium trioxide (Y_2O_3), with polyoxymethylene (POM) as binder. Parameters and settings of the CIM process are summarized in Table 1. The material properties can be found elsewhere [24].

The green parts were exposed to a de-binding process by nitric acid at 383 K, with a minimum loss of 17.5 %, and to a sintering cycle, performed in air (mild purge of air up to 873 K, sintering support Al_2O_3 with a purity of 99.6 %), consisting of the following typical steps:

- Heating from room temperature to 543 K with rate 3 K/min; hold on 1 hour.
- Heating from 543 K to 1773 K with rate 3 K/min; hold on 1 hour.
- Cooling from 1773 K to 873 K with rate 5 K/min.
- Furnace cooling.

The sintering process transformed the ceramic feedstock into polycrystalline yttria-stabilized tetragonal zirconia, with a typical composition of the final parts (commonly called sintered parts) of about

- 89 % of zirconium dioxide (ZrO^2)
- 5 % of diyttrium trioxide (Y_2O_3)
- 6 % of unspecified material(s) (not disclosed by the producer).

A considerable shrinkage is subsequent to the curing process (de-binding and sintering). It is normally accounted oversizing the mold dimensions. To obtain the desired sizes of the final sintered parts, the material producer specified an oversizing factor in the range 1.285 – 1.292.

Table 1. Parameters and settings of the ceramic injection molding process.

Parameter	Setting
Material type	ceramic feedstock (ZrO_2)
Barrel temperature /K	437-445
Mold temperature /K	413
Injection speed /cm^3 s^{-1}	8
Switch-over pressure /MPa	152
Cushion /cm^3	1.1
Packing pressure (pressure profile vs time)	0 s: 120 MPa, 1 s: 90 MPa, 2 s: 7.5 MPa
Total packing time /s	2
Machine	Arburg Allrounder 270 S 250-60 (15 mm)

Examples of a final product and of an intermediate part (after injection molding) are in Fig. 1. The micro mechanical component is a knocker, i.e. a critical movement part belonging to the mechanics of a watch. The knocker has to satisfy the requirements for movability. Hence, its functionality is entirely governed by the size of its features.

A sketch of the component, with a legend of the features of size defining its functionality, is shown in Fig. 2. The nominal values are summarized in Table 2 and Table 3. These values refer to the final dimensions (after the curing process). Table 4 instead reports the tolerances specified by the manufacturer, according to the ISO 2768-1 (class m) [25].

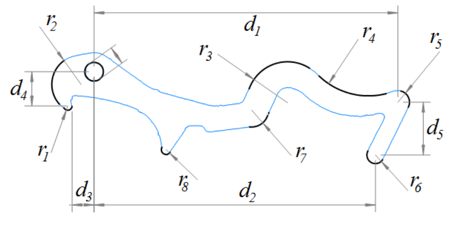

Fig. 2. Legend of the dimensions reported in Table 2 and Table 3.

Table 2. Nominal values in millimeters of the linear features specified in Fig. 2 (sintered components).

d_1	d_2	d_3	d_4	d_5	ϕ
7.939	7.515	0.612	0.984	1.438	0.40

Table 3. Nominal values in millimeters of the two-dimensional features specified in Fig. 2 (sintered components).

r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
0.08	0.75	1.06	1.82	0.30	0.22	0.40	0.10

Table 4. Tolerances in millimeters specified by the manufacturer, in the range of interest, according to ISO 2768-1, class m [25].

	Linear Dimension			Radii
Dimension	$d \leq 3$	$3 < d \leq 6$	$6 < d \leq 30$	$r \leq 3$
Tolerance	± 0.1	± 0.1	± 0.2	± 0.2

3. Metrology for quality assurance

The inspection of the quality assurance of the parts as well as the characterization of the production require consideration of the specific manufacturing process. As a general indication [2]-[3], to examine the accuracy of the curing process and the mold repeatability, twenty-five batches are normally selected, extracting five parts from each of them. Furthermore, the green parts are not stable. To characterize the curing process, the same period of time is to be considered after the CIM process. Even so, as already stated, the interest was to provide a method rather than to characterize the production itself. Hence, only five green and five sintered parts of the micro mechanical component were inspected. Sintered and green parts were chosen independently from each other. As a consequence, the analysis of this specific manufacturing process is to be considered in reproducibility conditions. All the features of size in Fig. 1, of both green and sintered parts, were measured using an optical coordinate measuring machine (OCMM – DeMeet 220). The uncertainty was evaluated according to the ISO 15530-3 [26], even though the substitution method was not applied (no correction of the average values performed). Average values, related expanded uncertainties for sintered parts and measured oversizing factors are in the Tables 5 to 8. Furthermore, a quality assessment was performed on the tolerance specifications assigned by the manufacturer (Tables from 2 to 4, Table 9 and Table 10).

Ideally, if the variability expressed by the expanded uncertainty is intrinsic to the production, the results can be considered an acceptable estimation. Conversely, if it is influenced by the measurement process the results cannot be related directly to the manufacturing process [1]-[3]. Indeed, the size of the features of the micro components ranges from several millimeters to tens of micrometers. This is challenging when the measurements are to be performed, although, it is particularly useful for examining dimensions and tolerance chains, at different scales, in the same process. Hence, in order to understand if the evaluation could rely on the measurement process, a capability ratio was evaluated considering two contributions, one related to the instrument U_{instr} and another one related to the production $U_{\mu PIM}$. Considering U_{instr} as the average of the uncertainties related to each single part (evaluated on seven repeated measurements) and U the one evaluated on all parts (five parts, seven repeated measurements each—see Table 5 and 6), $U_{\mu PIM}$ was estimated as [2]

$$U_{\mu PIM}^2 = U^2 - U_{instr}^2 \quad (1)$$

Eventually, an indication of the measuring process capability was given as the ratio between U_{instr} and $U_{\mu PIM}$ (see Table 11 and Table 12).

Table 5. Average values (first row) and expanded uncertainties (second row) of the sintered parts (lengths).

d_1 /mm	d_2 /mm	d_3 /mm	d_4 /mm	d_5 /mm	ϕ /mm
7.958	7.517	0.599	0.963	1.369	0.482
0.099	0.023	0.016	0.040	0.038	0.003

Table 6. Average values (first row) and expanded uncertainties (second row) of the sintered parts (radii).

r_1 /mm	r_2 /mm	r_3 /mm	r_4 /mm	r_5 /mm	r_6 /mm	r_7 /mm	r_8 /mm
0.073	0.765	1.122	1.799	0.272	0.215	0.397	0.098
0.021	0.077	0.021	0.163	0.046	0.051	0.020	0.019

Table 7. Measured oversizing factors of the linear features.

d_1	d_2	d_3	d_4	d_5	ϕ
1.283	1.291	1.308	1.280	1.300	1.256

Table 8. Measured oversizing factors of the two-dimensional features.

r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
1.619	1.230	1.213	1.341	1.525	1.293	1.282	1.394

Table 9. Lower and upper limits of the specification intervals for the linear features of the sintered parts.

d_1 /mm	d_2 /mm	d_3 /mm	d_4 /mm	d_5 /mm	ϕ /mm
7.798	7.373	0.512	0.899	1.287	0.465
8.119	7.661	0.686	1.026	1.450	0.498

Table 10. Lower and upper limits of the specification intervals for two-dimensional features of the sintered parts.

r_1 /mm	r_2 /mm	r_3 /mm	r_4 /mm	r_5 /mm	r_6 /mm	r_7 /mm	r_8 /mm
0.026	0.644	0.934	1.487	0.136	0.156	0.000	0.056
0.121	0.887	1.311	2.111	0.409	0.275	0.893	0.141

Table 11. Sintered parts (lengths): expanded uncertainties in micrometers related to the components U_{instr} and $U_{\mu PIM}$ and their capability ratio.

	d_1	d_2	d_3	d_4	d_5	ϕ
U_{instr}	26	5	11	15	18	2
$U_{\mu PIM}$	96	23	12	37	34	2
Ratio	27 %	20 %	97 %	41 %	55 %	78 %

Table 12. Sintered parts (radii): expanded uncertainties in micrometers related to the components U_{instr} and $U_{\mu PIM}$ and their capability ratio.

	r_1	r_2	r_3	r_4	r_5	r_6	r_7	r_8
U_{instr}	3	8	6	6	16	13	4	
$U_{\mu PIM}$	20	77	20	163	44	49	19	18
Ratio	15 %	10 %	33 %	4 %	36 %	28 %	23 %	32 %

4. Evaluation of the shrinkage and propagation of the uncertainty

The shrinkage due to the j_{th} green part was estimated for each dimension as the relative deviation from the corresponding average dimension of all sintered parts (stable parts), which was considered the reference for the achieved production:

$$\delta L_j = f\left(d_{x_j,g}, \overline{d}_{x,s}\right) = \frac{d_{x_j,g} - \overline{d}_{x,s}}{\overline{d}_{x,s}} \quad (2)$$

where

- $f\left(d_{x_j,g}, \overline{d}_{x,s}\right)$ is the shrinkage of the j_{th} green part.
- $d_{x_j,g}$ is a generic dimension of a green part.
- $\overline{d}_{x,s}$ is the average of a generic dimension of all sintered parts.

For evaluating the uncertainty of the shrinkage due to the sintering process, the uncertainty was propagated considering green and sintered parts correlated. According to the non-linear second order Taylor series of the shrinkage, the approximated expression used for the correlated quantities was [27]:

$$u_{x,j}^2 = \left(\frac{\partial f}{\partial d_{x_j,g}}\right)^2 \Delta d_{x_j,g}^2 + \left(\frac{\partial f}{\partial \overline{d}_{x,s}}\right)^2 \Delta \overline{d}_{x,s}^2 + $$
$$+2\frac{\partial f}{\partial d_{x_j,g}}\frac{\partial f}{\partial \overline{d}_{x,s}}\rho_{g,s}\Delta d_{x_j,g}\Delta \overline{d}_{x,s} \quad (3)$$

where

- $u_{x,j}$ is the uncertainty of the shrinkage propagated for the j_{th} green part.
- $\frac{\partial f}{\partial d_{x_j,g}}$ and $\frac{\partial f}{\partial \overline{d}_{x,s}}$ are respectively the partial derivative of the shrinkage with respect to the generic dimension of the j_{th} green part and the one with respect to the average value of the generic dimension considering all sintered parts (sensitivity coefficients).
- $\Delta d_{x_j,g}$ and $\Delta \overline{d}_{x,s}$ are respectively the variability of $d_{x_j,g}$ and the variability of $\overline{d}_{x,s}$ relative to the measurement process. These quantities were considered correlated with a degree of dependence given by the Pearson's correlation coefficient $\rho_{g,s}$. Furthermore, they are normally standard uncertainties. However, they were subjected to the purpose of introducing the method in § 5, where a different choice will be justified.

5. Formulation of specification intervals

The analysis of the shrinkage showed a different behavior of the linear and bi-dimensional features (see Fig. 3 and 4) which can be accounted more efficiently with respect to the specifications. At this purpose, the specifications are re-formulated as function of the variability of the production, mutually before (CIM process) and after (sintering process) the dimensional progression of the shrinkage.

The method allocates two-sided tolerance intervals in agreement with the guarded acceptance decision rule, defined in [4], with guard bands corresponding to a length parameter equal to the expanded uncertainty characterizing the production. In the same way, it is in agreement with the conformance zone defined in [5] and obtained reducing a specification zone by the expanded uncertainty characterizing the production.

Key point of the method is the assumption of propagating a coverage interval. As already stated regarding $\Delta d_{x_j,g}$ and $\Delta \overline{d}_{x,s}$, their values were fixed to non-conventional quantities in Equation (3) (propagation of the uncertainty of the shrinkage). In detail, the specifications (tolerances) were formulated propagating coverage intervals, evaluated according to a confidence level of the same amount of the desired conformance probability. In other words, this gives to the producer a risk of rejecting a part, in future evaluations, established according to a desired conformance probability controlled by the confidence level of the expanded uncertainty.

Having no specific indications, the choice was here a conformance probability of 95 %. Therefore, tolerance intervals could be estimated setting $\Delta d_{x_j,g}$ and $\Delta \overline{d}_{x,s}$ to the corresponding the expanded uncertainties, in the propagation of the shrinkage.

Considering several manufactured parts and several repeated measurements for each feature of each part, two sets of contributions can be propagated according to Equation (3): a propagated uncertainty u_{x_j} of each single part $d_{x_j,g}$ and a propagated uncertainty $u_{x,Av}$ of the average shrinkage $\overline{d}_{x,g}$ of all the green parts. Hence, the propagated uncertainty of the shrinkage of each green part (variability of the instrument) is square subtracted from the propagated uncertainty of the average shrinkage of all the green parts (total variability) to estimate the uncertainty of the process:

$$u_{proc,x_j}^2 = u_{x,Av}^2 - u_{x_j}^2 \quad (4)$$

Eventually, the specification limits were calculated as the quadratic average (average of the variances) of all the relative process uncertainties u_{proc,x_j}^2, extended to the desired confidence level by a coverage factor k:

$$U_{proc,x} = h_S \cdot k \cdot \left(\frac{1}{n}\sum_{j=1}^{n} u_{proc,x_j}^2\right)^{\frac{1}{2}} \quad (5)$$

where n is the number of green parts considered and $h_S \geqslant 1$ is a safety factor. It was added into the equation to satisfy the conformance interval with a certain margin (see conformance limits in Equations (7-a) and (7-b) below).

Hence, the specification intervals were calculated as

$$\overline{d}_{x,s} - U_{x,process} < d_{x,s} < \overline{d}_{x,s} + U_{x,process} \quad (6)$$

where it is $U_{x,process} = \overline{d}_{x,s} \cdot U_{proc,x}$ (not relative quantity).

Eventually, the conformance zones were identified by the following limits:

$$T_{x,L} = \bar{d}_{x,s} - U_{x,process} + U_{x,S} \qquad (7\text{-a})$$

$$T_{x,U} = \bar{d}_{x,s} + U_{x,process} - U_{x,S} \qquad (7\text{-b})$$

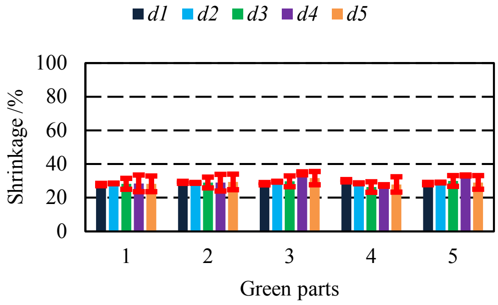

Fig. 3. Shrinkage (lengths). Error bars indicate the estimated u_{proc,x_j} .

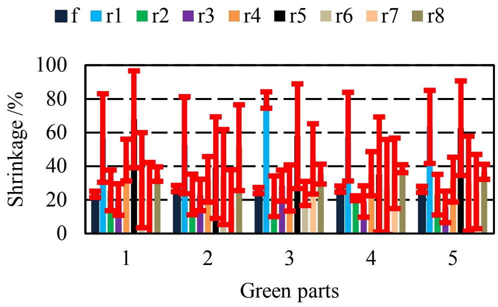

Fig. 4. Shrinkage (radii). Error bars indicate the estimated u_{proc,x_j} .

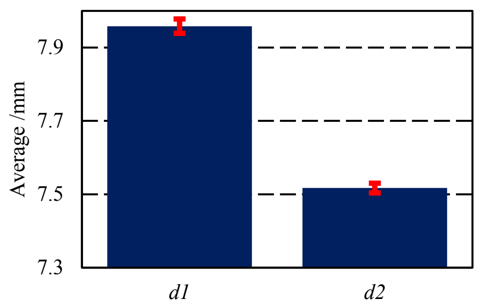

Fig. 5. Average values (columns) and specification limits (error bars).

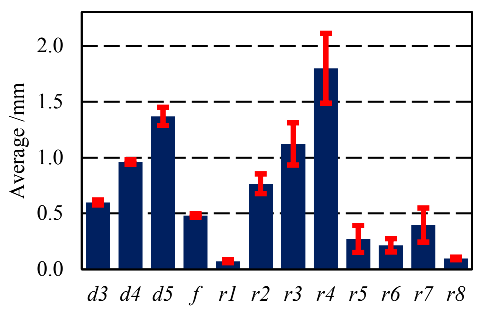

Fig. 6. Average values (columns) and specification limits (error bars).

where $U_{x,S}$ are the expanded uncertainties of the sintered parts, reported in Table 5 and Table 6.

6. Discussion

Considering a comparison between an average tendency of the manufacturing process, according to all the measured parts, with the specification intervals assigned by the manufacturer following the ISO 2768-1 [25], from the inspection of Table 5 to Table 10, the following can be said:

- The conformity to the specifications was proven for all the dimensions except d_4 and d_5.
- Neither the conformity nor the nonconformity to the specifications can be proven for d_4 and d_5.
- The oversizing factors are all outside the interval provided by the producer of the feedstock, except the one of d_2. However, the oversizing factors of d_1, d_4, d_5, r_6 and r_7 are very close to the nominal interval provided.

As a general trend for both green and sintered parts, the ratios between the expected uncertainties U_{instr} and the estimated uncertainties of the process $U_{\mu PIM}$ were larger than the 10 % stated by the *golden rule* of metrology, with few exceptions. It means that the measurement process influenced the evaluation of the uncertainty and, consequently, the conformance verification.

The reason regards the software equipped on the OCMM used. It identifies the geometrical entities in a specimen under measurement by an algorithm based on edge detection: an edge is recognized as sharp transition between a dark and a light area in the formed image. Unfortunately, many defects were found along the edges of the parts, which explain the poor performance of the instrument. The presence of defects on such manufactured parts is quite common. They could locally change and counteract the "light to dark" transition.

Even so, this poor outcome was concealed by the large tolerances stated by the producer: the lengths were specified with a precision of 1 µm, the radii with a precision of 10 µm whilst tolerances had a precision of 100 µm. This also emphasizes a lack of tolerance rules at the sub-mm scale. The conformity to the specifications, in fact, was almost verified mostly because the tolerance intervals were large and not adequate to the third and second decimal in the nominal values that were instead specified by the manufacturer.

The proposed method for the allocation of tolerances aimed to optimize the specifications considering the different impact of the shrinkage on the different features and, hence, accounting for them independently. The shrinkage was, in fact, constant for the linear dimensions while pretty uneven for the curved features (see Fig. 3 and Fig. 4). In this last case, a dependence of the shrinkage on the dimensions was also noticed, evidence of an anisotropic curing of two-dimensional features (see Fig. 4).

The impact of the measurement process on the investigation could not be considered negligible, mostly worse for the linear dimensions (Table 11 and Table 12). Hence, even though the linear features had tendency to constant shrinkage and not excessive uncertainty, the portion of variability due to the instrument inside the evaluated expanded uncertainty was large. An explanation for this contradiction was that linear dimensions were indirectly measured using the centers of curvature of the curved features as inputs, thus propagating systematic errors.

Samples of green and sintered components should be representative of the production for a fruitful formulation of the specifications. This means that a large number of samples are to be considered and, also, in ideal circumstances, the green parts should be measured before sintering and the same parts re-measured after the sintering process. In such way, the correlation among the parts would be fully exploited for a more accurate determination of the shrinkage uncertainty.

In addition, the influence of the instrument on the variability of the sintered parts (they are the reference in the propagation of the uncertainty) may affect the estimated variability of the green parts, leading to the formulation of wrong specifications.

7. Conclusion

The evaluation of the quality assurance in a micro powder injection molding production highlighted a constant shrinkage of the lengths and a non-uniform one of the radii that suggested a possible optimization of the specifications. A method for the synthesis of tolerance intervals was developed on purpose. It is based on the evaluation of the shrinkage and on an assigned conformance probability. It was demonstrated effective for dealing with non-linear shrinkage and for highlighting both non-optimal performance of the measurement instrument and unsuccessful measurement sessions.

The new developed shrinkage calibration procedure was applied and validated in the case of micro powder injection molding components. However, it is of general validity for any molding process, i.e., any process in which the material undergoes a change in dimensions from the mold cavity, due to a phase transformation.

Acknowledgements

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